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VLF Nitrogen Purge System

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13. ABSTRACT (*Maximum 200 words*)

This report summarizes the installation of Nitrogen Purge Systems and the accompanying dissolved oxygen sensor systems at the four water cooled very low frequency radio transmitting sites. These four sites differ in their physical plant and transmitting capabilities. To accommodate these differences, the size and configuration of the Nitrogen Purge Systems and Oxygen Sensing Systems are slightly different. The systems are composed of off-the-shelf items to allow for ease of maintenance and repair.

14. SUBJECT TERMS

Nitrogen purge system	Oxygen electrodes	Cupric oxide
Dissolved oxygen	Chemical corrosion	
VLF	Dissolved copper	

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VLF NITROGEN PURGE SYSTEM

INTRODUCTION

In 1980, NRL Chemistry Division personnel provided chemical analysis of a black solid forming on the electrode surfaces of very low frequency (VLF) radio transmitter power amplifier (PA) tubes. This solid was identified as cupric oxide, CuO, the buildup of which would inhibit system performance as a result of decreased cooling water flow rates and poorer heat transfer, eventually leading to overheating and failure of expensive power amplifier and intermediate power amplifier (IPA) tubes. In fact, inadequate or improper cooling due to scale or corrosion is considered the limiting factor in tube life [1]. The result of this apparently uncontrolled corrosion in the VLF transmitter cooling system was a study aimed at: (1) evaluation of the corrosion process; (2) design of a chemical cleaning method for the removal of corrosion products; and (3) development of a method to control or inhibit the corrosion process. The results of this study were documented in two detailed reports [2,3]. Briefly, the use of deaeration is a process by which the dissolved oxygen is physically removed from the water. The removal of dissolved oxygen from the cooling water prevents the formation of CuO. Sparging water with nitrogen is a simple and safe method to remove dissolved oxygen from the cooling water.

The intent of this report is to summarize the installation of Nitrogen Purge Systems and the accompanying oxygen sensor systems at the four water-cooled transmitter sites (Lualualei, Jim Creek, Harold E. Hold and Aquada) in one document for easy referral/filing purposes. Since the four sites differ in their physical plant and transmitting capabilities, the size and configuration of the Nitrogen Purge System and oxygen sensing system are slightly different. The oxygen sensing systems installed at the four sites are identical

For completeness, a short background on corrosion effects, criteria for corrosion prevention as related to VLF sites, and purge system recommendations are provided.

Manuscript approved July 29, 1999.

BACKGROUND

Corrosion in Water-Cooled Radio Transmitters

The power amplifier tubes used in most VLF radio transmitters dissipate such high power that they must be continuously cooled by water flowing at high velocities through the narrow channel located in the outer surface of the collector (or anode) [4]. This cooling water is periodically treated via circulation through a membrane purification system to maintain a high resistivity so as to prevent excessive scaling or corrosion. The transmitter is cooled by a closed recirculating system: a primary loop of treated pure water cools the power amplifier tubes, which in turn, is either air- or water-cooled in large heat exchangers (Appendix A, Figure 1).

Direct contact between the cooling water and the metal (copper) electrode surfaces ensures good heat transfer. However, corrosion of the metallic copper surfaces is evidenced by the buildup of black cupric oxide (CuO) in areas of high heat transfer to the coolant. The reaction which occurs at the copper surface is simplistically represented by equation (1) [where Δ = heat]:

$$(1) \qquad Cu^{o} + O_{2(g)} \xrightarrow{\Delta} CuO_{(s)}$$

The reaction drawn in equation (1) is in fact one of the two major sources of corrosion in a closed water-cooling system [1]. The resulting CuO corrosion product acts as a thermal insulator, thereby reducing heat dissipation, and accumulates in the small cooling passages, thus reducing the coolant water flow rate. The end result is premature tube failure.

Criteria for Corrosion Prevention

As outlined in a previous report [3], there are a number of potential mechanisms for corrosion prevention in water systems, the most viable for Navy VLF applications was determined to be the removal of the dissolved oxygen. This may be accomplished

either by direct treatment of the water or indirectly by introduction of an oxygen-free purge gas stream into the water to displace the dissolved oxygen. There are a number of methods which may be used for the removal of oxygen from flowing water; these are documented in reference 3. The criteria, which must be met by any implemented oxygen-control system, were identified as follows:

(1) Reduce the dissolved oxygen content of the cooling water from air-saturated levels (5 to 7 ppm) to less than 0.5 ppm by weight.

(2) Operable at temperatures and water flow rates found in Navy VLF cooling systems (38-52^{0}C; 120-140 gal/min (gpm) at 35 lbs/in^2 (psi) for PA circuits and 25 gpm at 40 psi for IPA circuits).

(3) Constructed of materials, which are compatible with transmitter conditions, such as stainless steel, copper, brass, and selected plastics and rubbers.

(4) Designed in such a manner that operation does not require the introduction of materials into the water that would interfere with the operation of the transmitter or which would result in the introduction of impurities. Unacceptable materials include any species, which might be subject to or contribute to electrolysis (i.e., ionic species), or corrosion-inducing materials (such as amines).

(5) Able to function independently and consistently be easily operated and maintained by station personnel, and low in cost.

Based on these criteria and preliminary experiments performed in 1985 at the Lualualei VLF facility, it was established that purging of the water system with an inert gas such as nitrogen, effectively displaced the dissolved oxygen and was successful in maintaining an essentially oxygen-free atmosphere.

NITROGEN PURGE SYSTEM

Prism Membrane Nitrogen System

A number of methods were identified to either supply or produce nitrogen gas for delivery into the VLF water system [3]. The method that met the criteria listed above is based on commercially available membrane technology. This technology utilizes hollow fiber, semi-permeable membranes to separate nitrogen gas from compressed air at a purity of >99% on a continuous basis and economically.

The nitrogen delivery unit is called the Prism Nitrogen System and is manufactured by Permea, Inc., a subsidiary of Air Products Company, St. Louis, MO. A schematic of the system is located in Appendix A, Figure 2. The following is a list of the Permea systems installed:

1) Lualualei, Permea Prism Alpha:
2) Jim Creek, Prism Nitrogen System Model 1300;
3) Harold E. Holt, Prism Nitrogen System Model 1500;
4) Aquada, Prism Nitrogen System Model 1100

The system installed at Lualualei was comprised of a compressor and a Prism Alpha separation unit. The other three systems installed were turnkey operations which consisting of an air compressor, the Prism membrane separator unit, and a nitrogen holding tank on a portable skid assembly. The Prism Nitrogen System is the most economical, reliable, low maintenance, on-site nitrogen supply unit available on the market today.

The Prism Nitrogen System separates nitrogen from ambient air (a mixture of nitrogen and oxygen with argon and other trace components) which is supplied via a compressor. In the turnkey systems, the air is compressed in an oil-lubricated rotary screw compressor (Kaeser Model) packaged with an integral air-cooled after cooler. The cooled air is supplied to a coalescing filter to trap water and oil which then feeds into the Prism Nitrogen System at approximately 190 psig.

The compressed feed air is passed through two filters with condensate traps to remove liquid water, oil and particulates that may be present before entering the separator

membranes. The cleaned air is heated in an electric heater to the normal operating temperature of 105-145 °F. If the temperature of the incoming air falls out of these limits, a red light alarm will be triggered. Once activated, the unit will automatically bypass all gas to vent. This is true also if the purity of the nitrogen generated is detected to be below the desired set purity. The alarm will be triggered and all of the gas (waste plus nitrogen) will be vented.

Under normal operation, the air is then fed into the separator membranes. The driving force for the separation of the gas stream is the difference between each component gas partial pressure on the inside of the membrane versus such component gas partial pressure outside the fiber. As the gas stream flows along the inner surface of the hollow fibers, each gas component responds to its partial pressure differential and permeates through to the outside. Oxygen and water permeate easily so that the outside gas stream, which is vented to the atmosphere, is rich in oxygen. Due to their lower solubility and diffusivity, gases such as nitrogen and argon permeate much slower through the membrane. Thus, the gas stream inside the fiber is enriched in nitrogen and exits through an outlet at 190 psi.

The turnkey systems have start-up instructions that vary slightly with the different model types. Essentially, the nitrogen generation systems are placed in an area that has good ventilation since the byproduct of the production of 99.9+% pure nitrogen is air that is enriched in oxygen. The system produces condensate and needs to have access to a drain to which the water can be dumped. The compressor pressurizes a compressed air holding tank (surge tank) which feeds the membrane separation unit. The nitrogen that is produced is feed to a nitrogen holding tank. The nitrogen holding tank pressures range from 150 to 190 psi. The nitrogen gas either goes into a manifold for distribution to the various sparging systems or is connected directly to the sparging unit through the pressure regulator.

Cooling Water Sparging Unit

The dissolved oxygen in the cooling water is removed with the use of a sparging unit. There is a sparing unit for each tank of cooling water. The sparging unit consists of:

1) 1/4" stainless steel tube and a stainless steel medium porosity gas frit;
2) flow meter;
3) pressure regulator.

Swage connectors are used to provide airtight unions. The sparging tube is approximately 4 feet in length. The sparging tube is fitted with a stainless steel gas frit and is placed into the holding tank and is accessed through the top of the tank using stainless steel bulkhead connectors. The flow meter is connected to the sparging tube to allow for a flow of 4-6 scfh of nitrogen into the water. The pressure regulator steps down the nitrogen feed pressure from the Prism Nitrogen generation system to 30 psi. All potential leak points are tested using snoop or some other appropriate leak detection fluid while under a pressure of nitrogen gas.

DISSOLVED OXYGEN SENSOR

In order to continuously monitor the concentration of oxygen dissolved in the VLF cooling water system, an oxygen sensor was installed in-line in the circulating water line directly prior to the purification loop. The sensor unit was placed in this position so the water purification system would remove any electrolyte from the water should the oxygen sensing system fail and leak electrolyte (i.e., potassium chloride (KCl)) into the cooling water. The total sensing system consists of a dissolved oxygen microprocessor transmitter (Model 4300), a dissolved oxygen electrode (12 mm, T-type), and necessary wire connections and cables. The manufacturer of the system chosen is Ingold Electrodes, Inc., a subsidiary of Mettler-Toledo Process Analytical, Inc., Wilmington, MA. The oxygen electrode and transmitter unit is identical at all the sites, unlike the nitrogen generation units, which are different models from the same manufacturer.

The principle components of the oxygen electrode are shown in Figure 3. The interior body of the electrode is inserted in a stainless steel electrode shaft. The interior of the electrode consists of a tubular silver anode, which surrounds a glass rod into which a platinum wire cathode is fused. A thermistor for the temperature compensation of electrode current is also incorporated in the glass rod.

The membrane cartridge, which is filled with electrolyte, is slid over the end of

the interior body. It is closed at the lower end with a fixed, reinforced, oxygen-permeable membrane, which separates the electrodes from the medium.

The oxygen sensor is based on polarographic oxygen measurements. The sensor basically consists of a working electrode (cathode), a counter electrode and reference electrode (anode). The electrolyte conductively connects the cathode and anode. When a suitable polarization voltage is applied between the anode and the cathode, oxygen is selectively reduced at the cathode surface as shown below:

Cathode reaction: $O_2 + 2H_2O + 4e^- \text{---->} 4OH^-$

Anode reaction: $4\ Ag + 4\ Cl^- \text{---->} 4\ AgCl + 4e^-$

These chemical reactions result in an electric current that is proportional to the oxygen partial pressure and, using appropriate algorithms, is be converted into concentration units.

Prior to any calibration, the oxygen electrode must be polarized. This can be done by one of two methods: (1) a separate polarizer module can be placed on the top of the electrode; or (2) the electrode can be attached to the transmitter. In either case, the electrode must be polarized for a minimum of six hours. Note that if the electrode is disconnected from either voltage source for more than 5 minutes, it must be repolarized. The length of repolarization time is proportional to the length of time that the electrode is disconnected from the voltage source. If unsure, the electrode should be left to polarize for the full six-hour period.

Only two types of calibration procedures need to be performed on this unit. The first is a temperature calibration and the second is a zero calibration. The specific operation of the transmitter keys is given in the manual in sections 4.3.1 and 4.3.2. It is recommended that these procedures be repeated only on an annual basis, unless there are indications of failure such as reading instability, etc.

To calibrate the sensing system to temperature, the polarized electrode was placed into a bucket of water, which had been removed, from the purification loop. A stand-alone thermometer was also placed into the bucket of water. When the reading on the thermometer stabilized, the transmitter temperature reading was adjusted to that reading.

To set the zero point of the sensing system, the electrode must be placed in an oxygen-free environment. Placing the electrode directly into the packet, which contains the Ingold Zeroing Gel, provides this. Note that gloves should be worn during handling of the gel. After the transmitter reading has stabilized (i.e., no change in the display reading for sixty seconds), then the transmitter can be set to zero as described in the manual [7], section 4.3.2. The Zeroing Gel can be used to calibrate multiple electrodes; however, it must be disposed of after 24 hours after being opened. The Zeroing Gel is a sodium hydroxide preparation. This gel must be diluted with 5 gallons of water before disposal. There should be no reason to rezero an electrode unless something is replaced, such as electrolyte or membrane module.

NITROGEN GENERATION AND OXYGEN SENSING SYSTEMS AT THE SPECIFIC SITES

The guiding principle for selection of the nitrogen generating system was that a threefold capacity system be designed as a safety factor in order for the system to continue to operate even under less than optimum conditions. With this in mind, the systems were purchased that incorporated the safety margin necessary to maintain operations in the event of system capacity issues.

The Permea Prism Alpha system installed at Lualualei is shown in Figure 4a. This system was not a turnkey system. A separate compressor unit (Figure 4b) was purchased to supply the comprised air to the membrane system. The maximum nitrogen produced at the 99.9% purity level for this unit is 93 scfh. The oxygen electrodes and oxygen transmitters are shown in Figure 4c. The view is of the two oxygen electrode systems that are monitoring the two PA tanks. There is a third oxygen electrode system which monitors the IPA tanks (not shown) located on the adjacent wall. The transmitting units are the black displays mounted at the top of the figure. The oxygen electrodes are located in the bottom center and lower right of the figure. The electrodes are held in brass tees that are placed in the water purification loop just before the Barnestead filters.

Figure 5a is a view of the Model 1300 installed at Jim Creek. This is a turnkey unit that is comprised of a membrane separation unit, air surge tank, compressor and nitrogen storage tank. This system has an output of 62 scfh at a purity of 99.9%. Figure 5b shows the location of the single oxygen electrode system located above the Barnstead filter unit. The two PA tanks are monitored using this single oxygen system. This is the only site at which there is not an oxygen monitoring system for each of the PA tanks.

Figure 6a shows a front-on view of the Model 1500 (now Model 2300) Prism nitrogen system installed at Harold E. Holt. The physical constraints of equipment on either side of the system did not allow for a side view. This unit is slightly larger than the unit installed at Jim Creek. It also is a turnkey unit which produces 105 scfh at a purity of 99.9%. Figure 6b shows the typical oxygen transmitting system installed at each of the four PA tanks and the two IPA tanks (total of 6 units).

The Prism Nitrogen system Model 1100 installed in Aquada is shown in Figure 7a. Again this is a turnkey installation. However, since the nitrogen generation unit produces only 21 scfh of nitrogen at 99.9%, the demand for compressed air is much less than at Jim Creek and Harold E. Holt. Consequently, the air surge holding tank is nor included in the turnkey design. The nitrogen storage tank can be seen in the background. Figure 7b shows the placement of the single oxygen sensing unit. Again it is placed up stream of the Barnstead filter unit.

In Figure 8 is shown a typical installation for the nitrogen sparging system. The nitrogen pressure feed to the flow meter and then to the sparging unit inside the tank is controlled by a single stage regulator. In some installations, the sparging unit is located on the access door located on the top of the tank. In other cases, it is located adjacent to the access door. The sparging tube needs to located such that it is within an arm's reach of the tank opening so it can be attached to the bulkhead union which is installed in the top of all the tanks.

Table I in Appendix A lists all the Permea Prism Nitrogen generating units. The Alpha Prism unit installed at Lualualei is not listed since it was one of the initial systems that were produced and was not a turnkey system. The design specifications presented in the table allow one to project the unit's footprint into possible installation sites and determine the feasibility of using different capacity units. This table was taken from the

literature provided by Air Products. Larger capacity models up to 100,000 scfh are available.

1.4 REFERENCES

[1] Varian, Application Engineering Bulletin (AEB-17A).

[2] NRL Ltr Rpt 6180-104:KASH: August 1990.

[3] NRL Ltr Rpt 6180-105:AME: April 1990.

[4] Syrett. B.S., **1977,** *Corrosion,* 257.

APPENDIX A

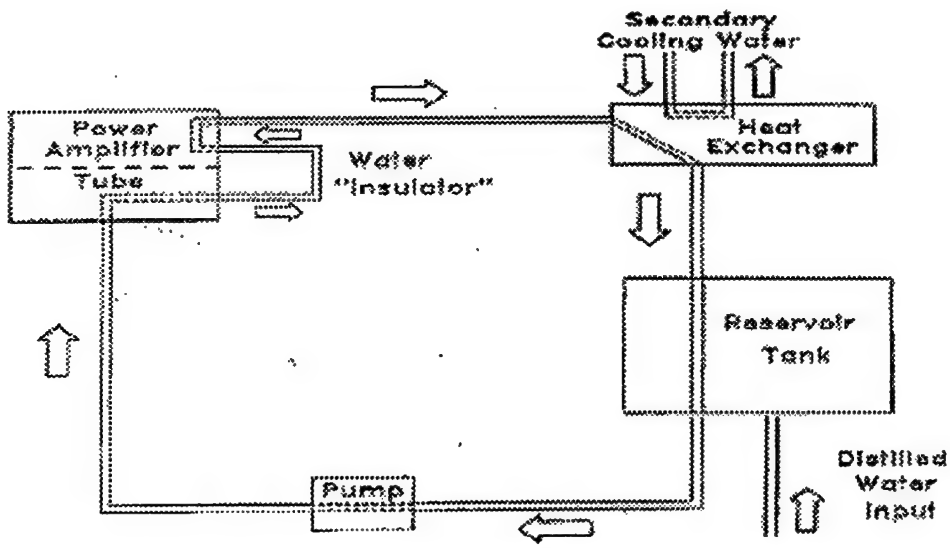

Figure 1. Portion of typical VLF transmitting cooling system

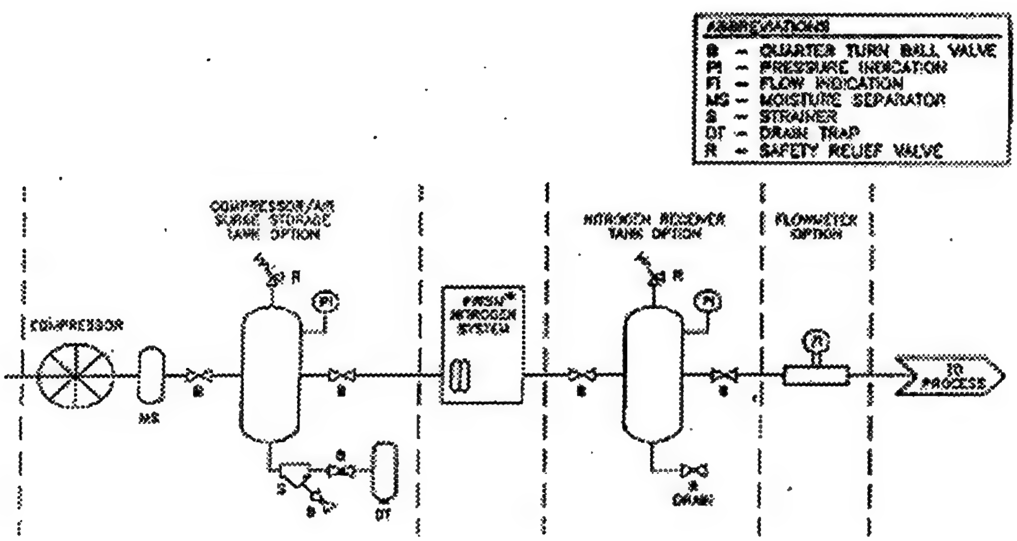

Figure 2. PRISM membrane cabinet system schematic

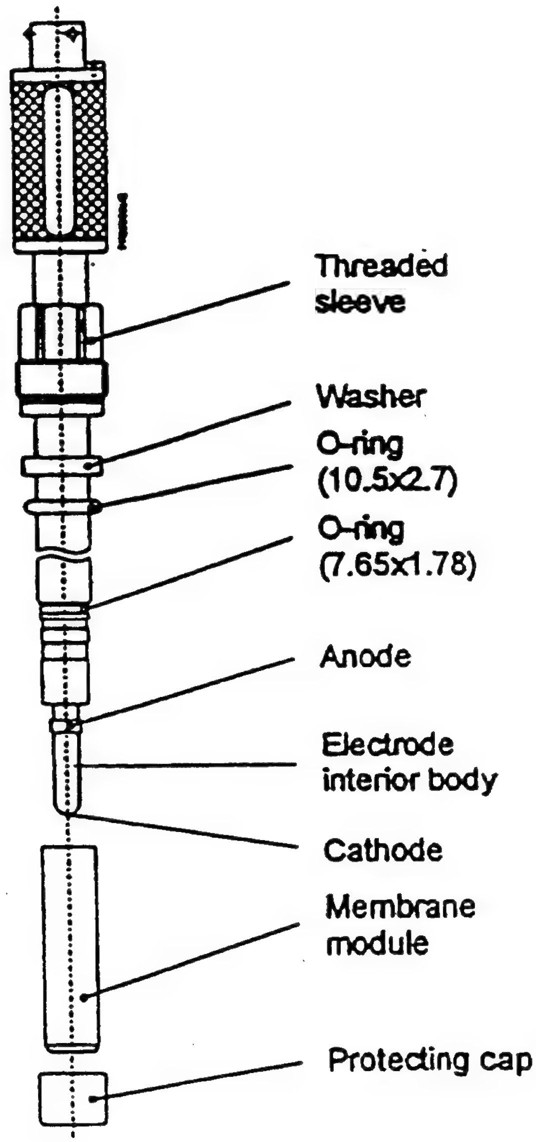

Figure 3. INGOLD dissolved oxygen electrode.

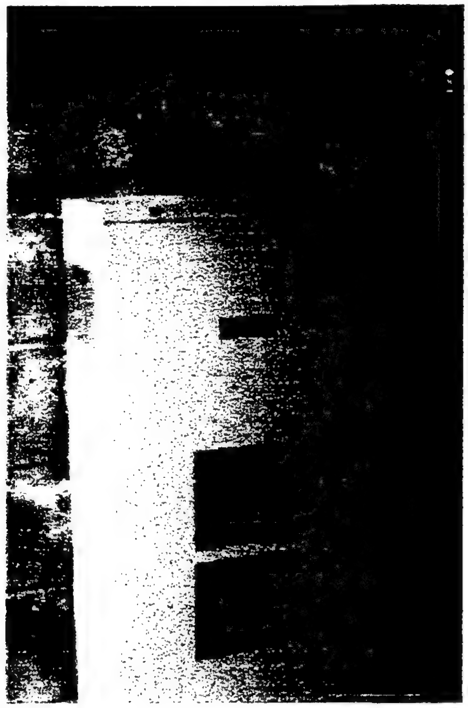

Figure 4a. Permea Prism Alpha installed at Lualualei.

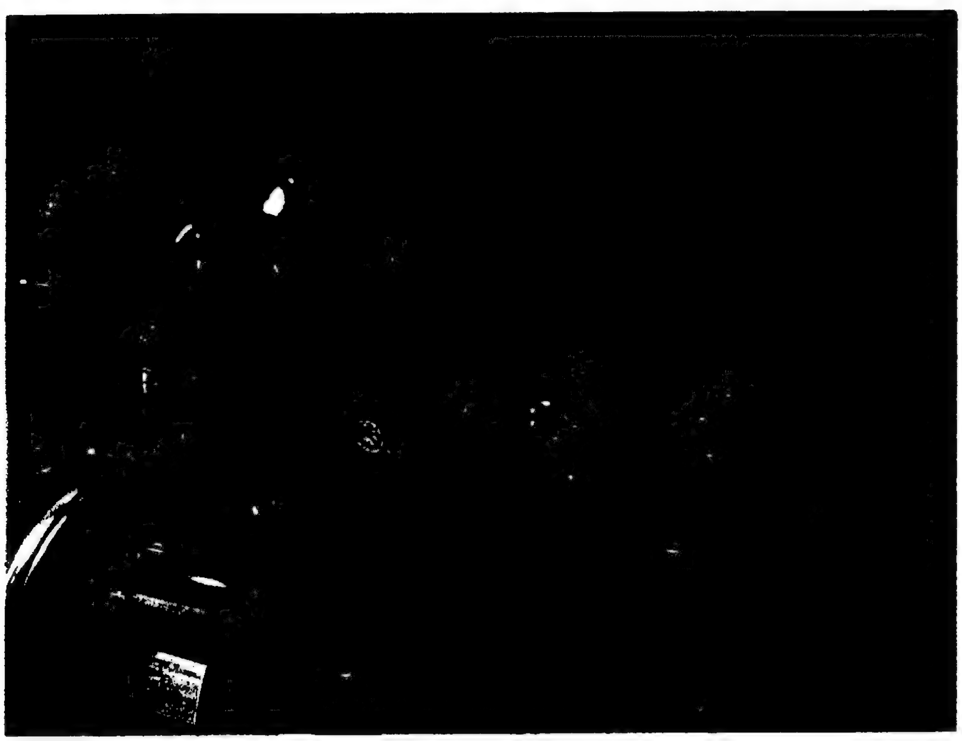

Figure 4b. Compressor used with Permea Prism Alpha system in Lualualei.

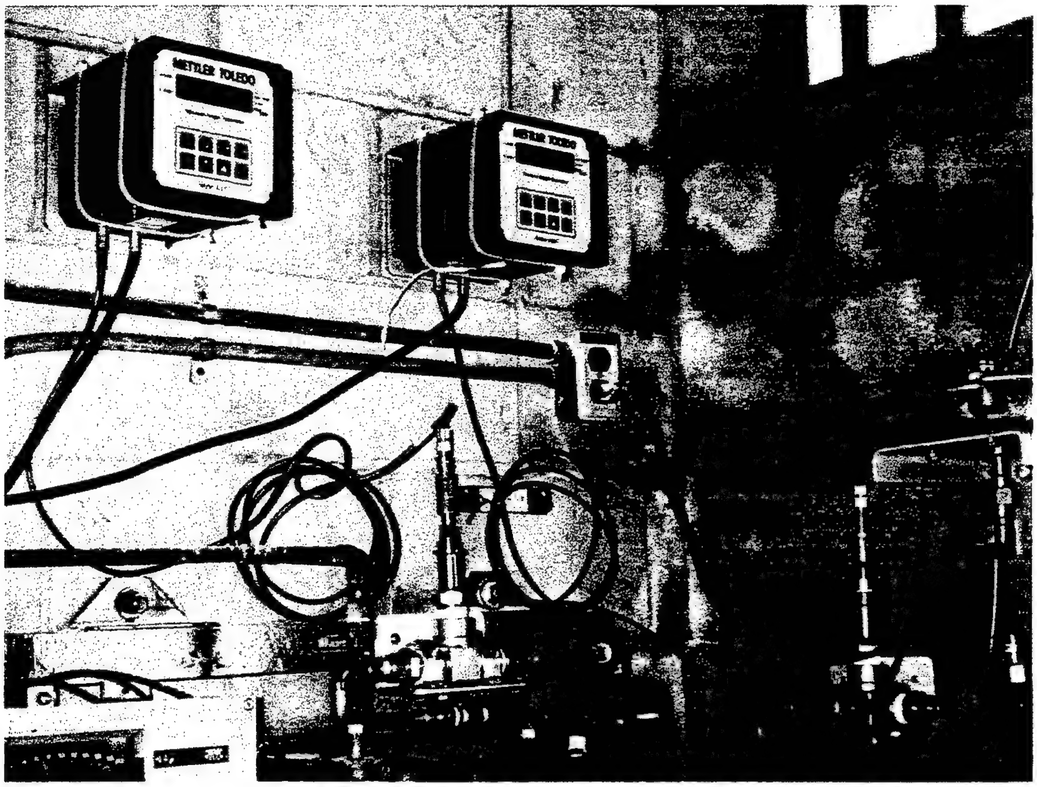

Figure 4c. Mettler Ingold oxygen transmitters and oxygen electrodes in brass tee holders installed at Lualualei.

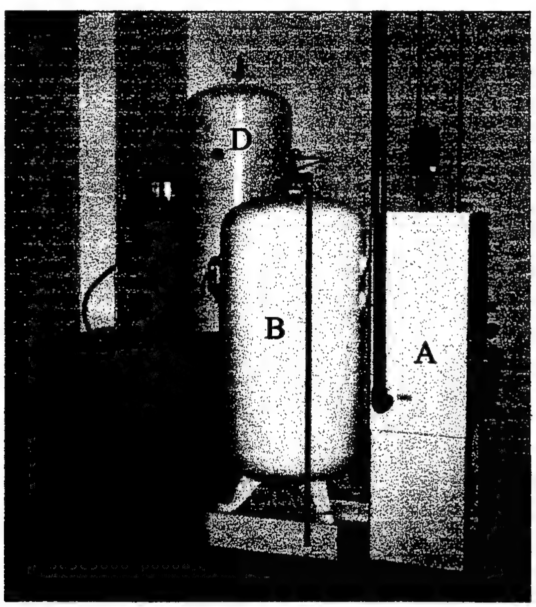

Figure 5a. Prism Nitrogen system Model 1300 installed at Jim Creek.
A = Nitrogen membrane separation unit; B = Air surge tank;
C = Compressor unit; D = Nitrogen storage tank.

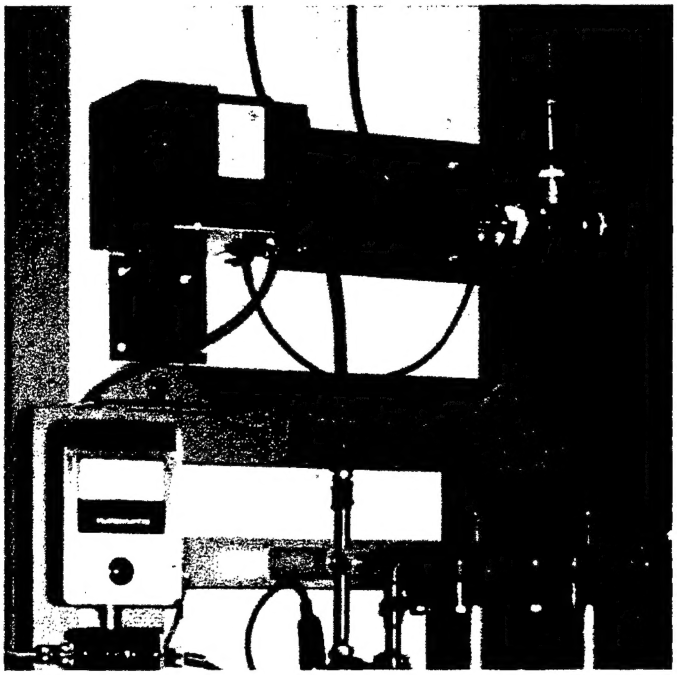

Figure 5b. Mettler Ingold oxygen transmitter and oxygen electrodes in brass tee holder installed at Jim Creek.

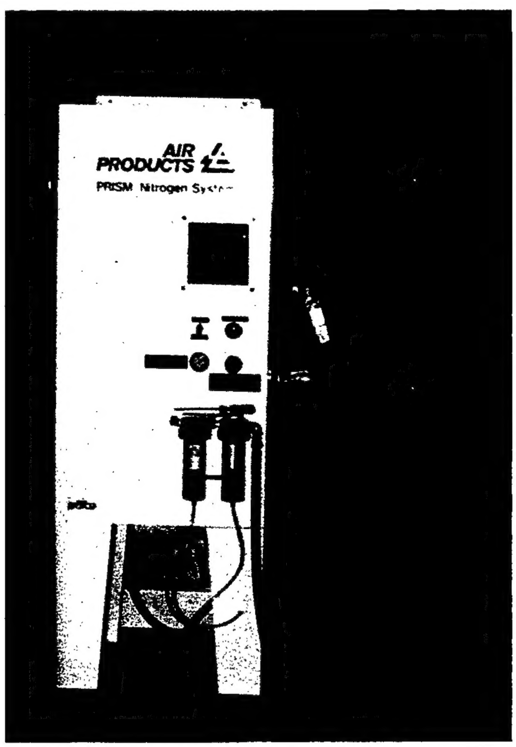

Figure 6a. Prism Nitrogen system Model 1500 installed at Harold E. Holt. Same configuration as Model 1300 installed at Jim Creek.

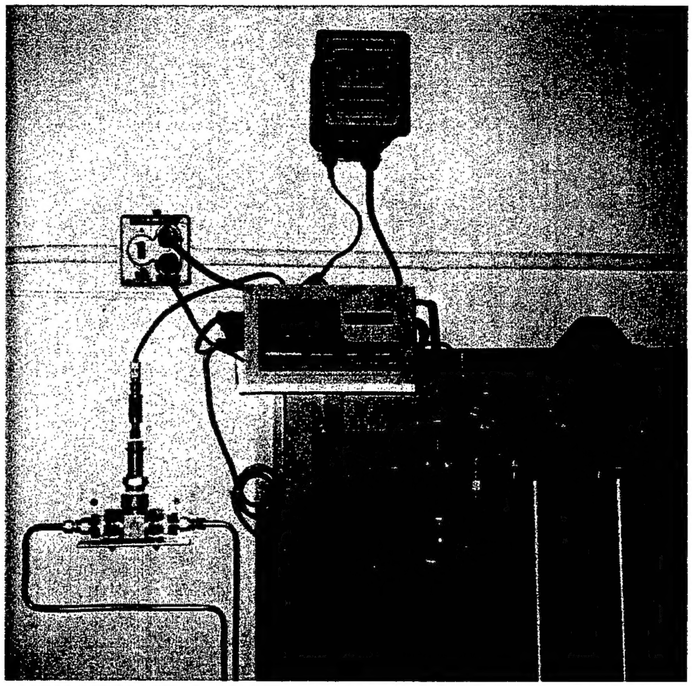

Figure 6b. Transmitter unit and oxygen electrode in brass tee holder installed at Harold E. Holt.

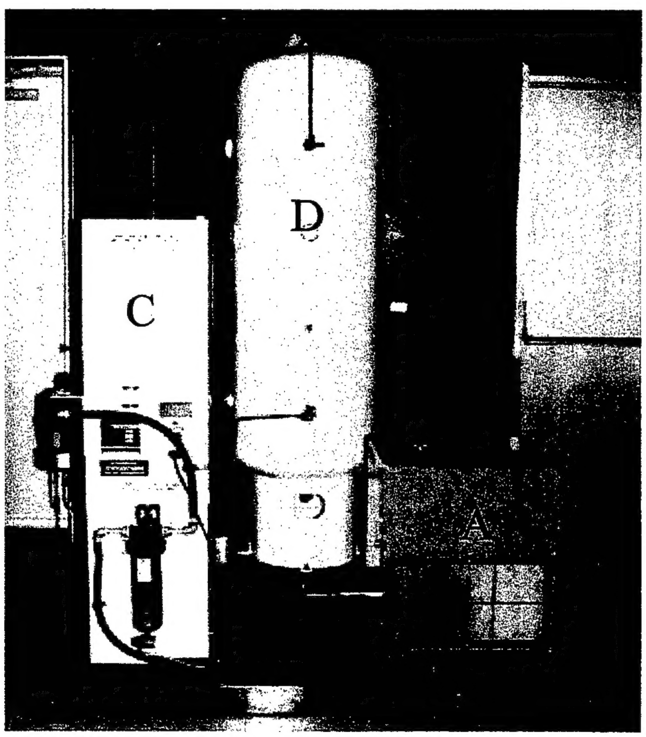

Figure 7a.. Prism Nitrogen system Model 1100 installed at Aquada. Since the system is of reduced capacity, the air surge tank (B) shown in figure 5a is not present.

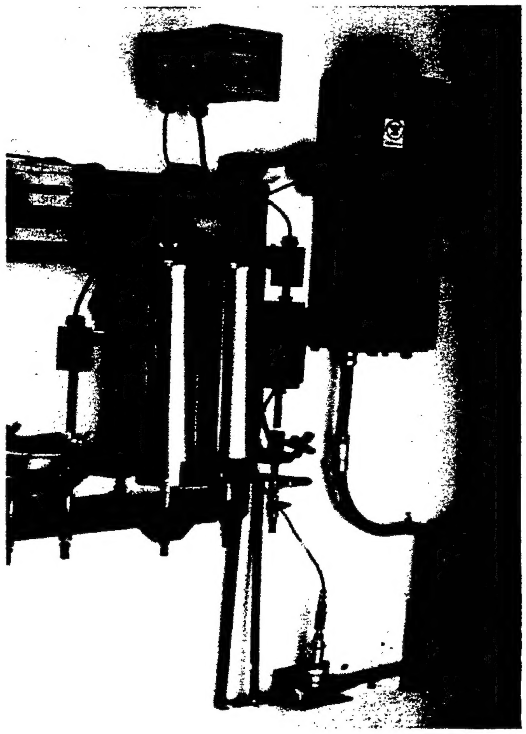

Figure 7b. Transmitter and oxygen sensor installed at Aquada

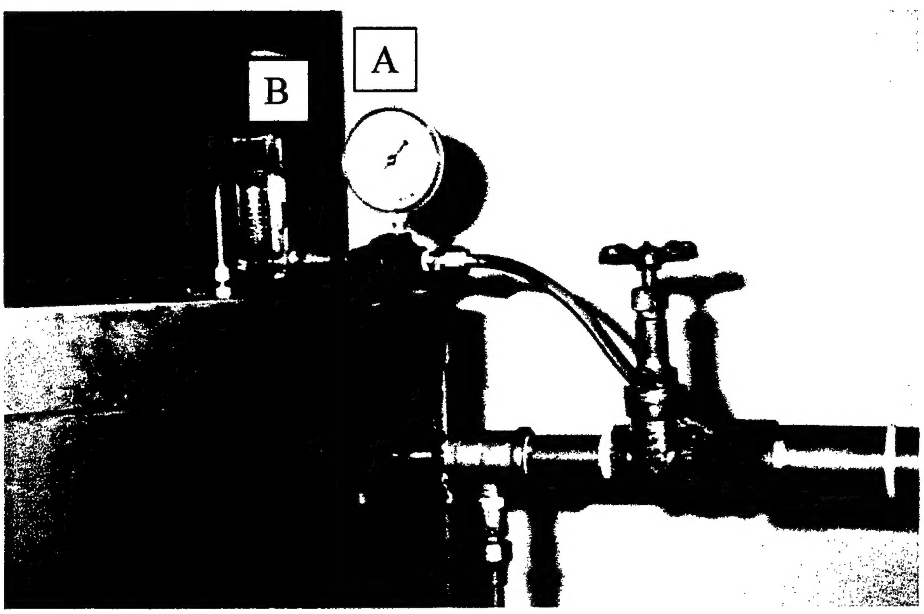

Figure 8. Sparging unit located on PA tank at Harold E. Holt showing the reducing regulator (A) and the flow controller (B). The components A and B can be located on the top of the holding tanks or in a manifold mounted on a nearby wall.

PRISM® Systems Operating Data

(Supply Air Pressure: 190 PSIG)

Purity > Model	99.9	99.5	99.0	98.0	97.0	96.0	95.0
1100	21	37	50	71	91	111	133
1200	41	74	101	143	182	223	266
1300	62	111	151	214	274	334	399
1400	83	149	202	285	365		
2300	105	188	256	361	462	565	674
2400	140	251	341	482	616	753	898
2500	175	314	427	602	770	941	
2600	209	377	512	722			

Larger standard systems available for flow rates up to 100,000 SCFH.

Design Specifications

PRISM Systems	Series 1000	Series 2000
Ambient air temperature	35–105°F (1.6–40.6°C)	35–105°F (1.6–40.6°C)
Nitrogen outlet	1/4" NPT female	3/8" NPT female
Nitrogen product pressure	170 to 185 psig	170 to 185 psig
Nitrogen dew point down to	–90°F (–67°C)*	–90°F (–67°C)*
Power (PRISM membrane)	110v, 60Hz, 550 watts	110v, 60Hz, 1000 watts
Power (air compressor)	230v/460v, 3 phase, 60Hz	230v/460v, 3 phase, 60Hz
PRISM skid (size)	76"L x 33"W x 72"H	76"L x 37"W x 72"H
PRISM skid (weight)	1,000–1,200 lbs.	1,250–1,500 lbs.
Nitrogen tank skid (size)	33"L x 33"W x 96"H	33"L x 33"W x 96"H
Nitrogen tank skid (weight)	450 lbs.	450 lbs.

*At atmospheric pressure.

Table I. Listing of all the Permea Prism Nitrogen generating systems specifications.